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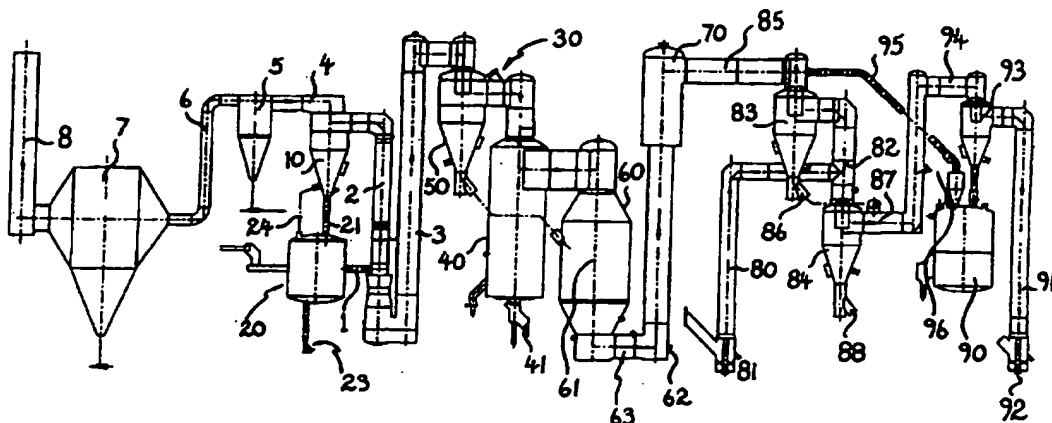
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(54) Title: PROCESS FOR THE PRODUCTION OF ALPHA ALUMINA



(57) Abstract

The present invention provides a process for the production of alpha alumina, the process comprising the steps of: (a) drying and dehydrating alumina in a dehydroxylyzation zone to remove residual water; (b) heating the dehydrated alumina in a calcining zone in the presence of hydrogen fluoride gas to a temperature above that in the dehydroxylyzation zone to commence transformation of the alumina to alpha alumina; and (c) holding the heated alumina in a holding zone for sufficient time and at a sufficient temperature to complete the transformation of the alumina to alpha alumina; wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone. Furthermore, the process of the invention preferably includes the additional step of adding steam, preferably excess steam, directly to the  $\text{AlF}_3$  at that point to ensure that there is virtually immediate generation of excess HF.

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## "PROCESS FOR THE PRODUCTION OF ALPHA ALUMINA"

### Brief Summary Of Invention

The present invention relates to a process for the production of alpha alumina and also to apparatus suitable for the operation of that process. The process  
5 preferably produces 100% alpha alumina, or at least produces alumina that is as close to 100% alpha alumina as is able to be measured by standard techniques.

### Background

Alpha alumina is a crystalline form of alumina generally having high hardness and only a weak tendency to hydrate, and is widely used as a raw material for  
10 abrasives, insulating bodies, sintered products, plasma spraying materials, and filler (among other things). Alpha alumina is most often produced by calcination of a technical grade aluminium hydroxide such as that produced by the Bayer process.

However, mere calcination will usually not convert enough of the alumina  
15 produced to alpha alumina. In this respect, alumina may be transformed to its alpha phase by several processes, the most common of which is to hold the alumina for an extended period at a temperature in the range of 1450 to 1500°C, although this temperature may be reduced to about 1250°C by the addition of a mineralizing agent such as aluminium fluoride ( $\text{AlF}_3$ ).

20 Two equipment types are most often used for the commercial production of alpha alumina. They are the kiln (rotary or tunnel) and the flash calciner, with the majority of alpha alumina (over 90%) being produced in kilns.

In kiln production, aluminium hydroxides or leached calcined alumina is de-liquored on a filter and then fed into the top (or cold) end of a rotating or  
25 tunnel kiln. Aluminium fluoride ( $\text{AlF}_3$ ) in a solid form is added with the feed at a rate proportional to the feed rate, and the alumina is heated by energy transfer

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from outgoing gases which are the products of combustion, with oil or gas burnt at the bottom (or hot) end of the kiln. The alumina is conveyed counter-current to the gases, achieving maximum temperature in a (so called) combustion zone of the kiln.

- 5 Upon discharge from the kiln, the alumina is cooled either directly with air or indirectly with water. The waste gases pass through a dust removal stage or stages, usually multi-cyclones and an electrostatic precipitator (ESP), before going to atmosphere.

- This technology suffers from several drawbacks, including low throughput per  
10 unit capital employed, poor heat distribution within the kiln, inefficient energy usage and high maintenance requirements. Other problems associated with the process are the high attrition index of the product with the potential for excessive dust losses to the atmosphere, causing environmental problems and producing a yield loss, and the presence of hydrogen fluoride (HF) gases in the waste gas  
15 stream resulting from the breakdown of  $\text{AlF}_3$ .

Furthermore, as the  $\text{AlF}_3$  is typically added to the alumina feed as it enters the process, usually on the filter during the deliquoring stage, there are generally high usage rates of  $\text{AlF}_3$  due to there being some losses with the waste gases as the  $\text{AlF}_3$  enters the process stream.

- 20 In flash calciner production, aluminium hydroxide or leached alumina feed is de-liquored and washed prior to delivery into the flash drying section of the calciner,  $\text{AlF}_3$  is added to the filter as is done with kiln operations. The material is heated and held at high temperature for approximately 30 to 50 minutes, following which the alumina is cooled by air in a flash cooling section prior to  
25 discharge to a surge bin.

Steam and air are generally used to fluidise the material in the holding vessel and excess air to combustion requirements is usually added to the flash cooling

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section to achieve the desired cooling of the product, while water is added in the cross-over between the flash drier and furnace to control stack temperatures.

The disadvantages with this process are defined in terms of excess fluoride emissions to the atmosphere, lack of uniformity in the product, poor efficiency of  
5 fluoride usage, and yield losses due to the high dust levels in the process.

### Summary Of The Invention

The present invention provides a process for the production of alpha alumina, the process comprising the steps of:

- 10 (a) drying and dehydrating alumina in a dehydroxylation zone to remove residual water;
- (b) heating the dehydrated alumina in a calcining zone in the presence of hydrogen fluoride gas to a temperature above that in the dehydroxylation zone to commence transformation of the alumina to alpha alumina; and
- 15 (c) holding the heated alumina in a holding zone for sufficient time and at a sufficient temperature to complete the transformation of the alumina to alpha alumina;

20 wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone.

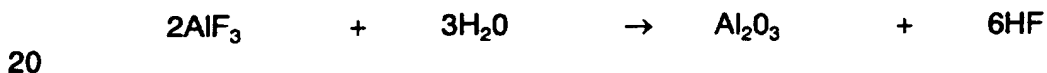
The alumina is preferably dried and dehydrated in the dehydroxylation zone to remove substantially all of the residual water. In this respect, it will be appreciated that the term drying refers generally to the removal of free water from the surface of the alumina, while the term dehydrating refers generally to  
25 the removal of water of hydration from the alumina. Thus, if the feed alumina is leached smelting grade alumina (the preferred feed alumina) there may be up to

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about 25% free water (on a wet basis) and possibly about 1% rehydrated water, substantially all of which is preferably removed, whereas an aluminium trihydrate as the feed alumina will contain up to about 10% free water and in the order of 35 to 40% rehydratable alumina, the drying and rehydrating again preferably removing substantially all of this water.

The temperature in the dehydroxylation zone may be in the range of 100°C to 800°C, although a temperature of about 150°C is preferred. In this respect, it will be appreciated that the most preferred temperature for this zone will vary according to the moisture content of the alumina feed and thus according to the moisture removal requirements.

The heating in step (b) of the dehydrated alumina from step (a) is preferably conducted in a furnace. The aluminium fluoride, together with steam, is preferably injected into, for example, the bottom of the furnace, at or near the entry to the calcining zone. In this respect, the temperature in the bottom of the furnace will generally be above 500°C, but will preferably be above about 1000°C. Under these conditions, pyrohydrolysis of the  $\text{AlF}_3$  occurs very rapidly to form hydrogen fluoride gas. Indeed, hydrogen fluoride gas (HF) is generated in the calcining zone by the following reaction:



The HF produced then acts as a mineralizing or catalytic agent which assists in the transformation of alumina to alpha alumina.

The production of the HF gas at or near the entry to the calcining zone, such as immediately prior to entry into the furnace, ensures that there is a greater concentration of this gas in the calcining zone where the alumina crystal transformation takes place. Furthermore, the process of the invention preferably includes the additional step of adding steam, preferably excess steam, directly to the  $\text{AlF}_3$  at that point to ensure that there is virtually immediate generation of excess HF. Thus, the process is not forced to wait for the passage of suitable

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levels of moisture in the gas to ensure that sufficient levels of HF gas are generated, excess levels being generated immediately prior to exposure of the alumina to the high temperatures in the furnace, providing beneficially higher efficiency and conversion rates for the transformation of alumina to alpha  
5 alumina.

The alumina thus then transfers to the calcining zone of step (b) with the excess levels of HF, wherein the alumina is heated in the furnace in the presence of that hydrogen fluoride to a temperature elevated above that reached in the dehydroxylation zone. Preferably, the alumina is heated above 1000°C, but  
10 more preferably the temperature range is 1120 to 1280°C, with a particularly preferred temperature being about 1200°C. The residence time for the alumina in the furnace is preferably short, for instance in the range of 10 to 100 seconds.

Preferably, the holding zone of step (c) includes, as at least a part thereof, a fluidized bed within which the heated alumina may be maintained for the  
15 sufficient time and temperature referred to. In this respect, the heated alumina is preferably maintained in the fluidised bed for about 1 to 45 minutes at a temperature in the range of 1200°C to 1300°C, or more preferably for about 30 to 45 minutes (ideally about 40 minutes) in a range of 1250°C to 1260°C.

In a preferred form of the invention there is provided the further step of  
20 contacting waste fluoride-containing gases with an agent or agents capable of reacting with the fluorides to remove them from the waste gases. Agents capable of reacting with fluorides will be known to a person of ordinary skill in the art. However, a particularly preferred agent is smelter grade alumina dust. The inclusion of this further step assists in ensuring that fluoride emissions to the  
25 atmosphere (which are heavily regulated) are within acceptable levels.

Thus, the process of the present invention is preferably also provided with a means to maintain levels of both particulate dust and hydrogen fluoride concentrations below environmentally acceptable levels. The dust produced by

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the process of the present invention is extremely fine and has a high electrical resistivity. That is, it is very difficult to capture in, for example, an electro-static precipitator (ESP), and once captured, is very difficult to remove from the collecting plates. Similarly, without an efficient cleaning mechanism to remove  
5 hydrogen fluoride from the outgoing gas stream, excessive quantities of the gas would be lost to the atmosphere.

As high surface area alumina will adsorb up to 4% of fluoride (by weight), and therefore act as a very effective "scavenging" agent for fluoride gas, the process of the present invention preferably includes the step of mixing a stream of  
10 smelter grade alumina dust with the waste gases from the dehydroxylation zone. That dust acts as a scrubbing medium to remove the fluoride from the waste gas stream. Thus, when captured on the collector plates on the ESP, the comparative low resistivity of the mixed dust permits easier removal of the dust layer from these plates.

15 Preferably, waste gases from each of the zones in the present invention are recycled in other zones of the process. Outgoing gases from the dehydroxylation zone are preferably combined with alumina scavenging dust for fluoride removal then passed into a dust collection device. Outgoing hot gases from the calcining zone may be used in the dehydroxylation zone to  
20 remove residual water from the alumina prior to entry into the calcining zone. Outgoing gases from the cooling zone may also be pre-heated and employed in the calcining zone. By recycling waste gases in this manner, maximum use may be made of the energy used to heat the gas prior to its release from the process. Furthermore, efficient control over fluoride usage may be achieved prior to its  
25 capture in the electrostatic precipitator.

In a further preferred form of the invention there may be provided an additional step which comprises cooling the alpha alumina produced in step (c) in a cooling zone where the temperature of the alpha alumina is reduced to less than 200°C prior to discharge from the process. The cooling zone preferably comprises a  
30 series of cyclones where the alumina is partially cooled, followed by final cooling



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in a flash cooler where the alumina may be held in a fluidized bed. That bed is preferably created using a high pressure-drop tuyere-type distributor plate, as alpha aluminas are generally difficult to fluidize. Indeed, such a distributor plate is preferably also used in the fluidised bed of the holding zone of step (c).

5 Thus, the present invention further provides a process for the production of alpha alumina, the process comprising the steps of:

- (a) drying and dehydrating alumina in a dehydroxylation zone at a temperature in the range of 100°C to 800°C to remove substantially all residual water ;
- 10 (b) heating the dehydrated alumina in a calcining zone within a furnace, in the presence of hydrogen fluoride gas, to a temperature in the range of 1120°C to 1280°C to commence transformation of the alumina to alpha alumina;
- 15 (c) holding the heated alumina in a holding zone within a fluidised bed for 30 to 45 minutes at a temperature in the range of 1200°C to 1300°C to complete the transformation of the alumina to alpha alumina; and
- 20 (d) cooling the alpha alumina in a cooling zone where the temperature of the alpha alumina is reduced to less than 200°C prior to discharge ;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone, excess steam being added to the aluminium fluoride at that point to ensure the immediate generation of excess hydrogen fluoride.

25 Further still, the present invention also provides a process for the production of alpha alumina, the process comprising the steps of:

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- 5 (a) drying and dehydrating alumina in a dehydroxylation zone at a temperature in the range of 100°C to 800°C to remove substantially all residual water, thereby producing dehydrated alumina and waste gases, said waste gases being mixed with a stream of smelter grade alumina dust to remove fluoride introduced by the outgoing hot gases from step (b);
- 10 (b) heating the dehydrated alumina in a calcining zone within a furnace, in the presence of hydrogen fluoride gas, to a temperature in the range of 1120°C to 1280°C to commence transformation of the alumina to alpha alumina, and thereby generating outgoing hot gases for use in the dehydroxylation zone of step (a);
- 15 (c) holding the heated alumina in a holding zone within a fluidised bed for 30 to 45 minutes at a temperature in the range of 1200°C to 1300°C to complete the transformation of the alumina to alpha alumina;
- (d) cooling the alpha alumina in a cooling zone where the temperature of the alpha alumina is reduced to less than 200°C prior to discharge, thereby producing a cooled alpha alumina product and outgoing gases ; and
- 20 (e) heating the cooling zone outgoing gases for return to the calcining zone of step (b) ;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone, excess steam being added to the aluminium fluoride at that point to ensure the immediate generation of excess hydrogen fluoride.

25

The present invention also provides apparatus for the production of alpha alumina, the apparatus comprising :

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- 5 (a) means for dehydrating alumina in a dehydroxylation zone to remove residual water;
- (b) means for heating the dehydrated alumina in a calcining zone in the presence of hydrogen fluoride gas to a temperature above that in the dehydroxylation zone to commence transformation of the alumina to alpha alumina; and
- (c) means for holding the heated alumina in a holding zone for sufficient time and at a sufficient temperature to complete the transformation of the alumina to alpha alumina;
- 10 wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone.

The means for heating the dehydrated alumina is preferably a furnace having a base at which hot gases and the dehydrated alumina enter. The temperature of  
15 the material entering the furnace will thus be lower than the temperature within the calcining zone within the furnace, and the furnace is preferably configured so as to be able to receive  $\text{AlF}_3$  at or near the base thereof so as to allow the pyrohydrolysis reaction referred to above to occur immediately before the gases and alumina enter the calcining zone.

- 20 The means for holding the heated alumina preferably includes a fluidised bed within which the alumina may be held for the sufficient time and temperature referred to above. The fluidised bed preferably includes a tuyere-type distributor plate at the bottom thereof to assist in fluidising the alpha alumina.

#### Detailed Description Of The Invention

- 25 The present invention will now be described in detail by way of example with reference to the accompanying flow-sheet wherein Figure 1 is a schematic

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representation of a fluid flash calcination system for carrying out the process of the invention.

Feed alumina in the form of leached smelter grade alumina is pumped from a leach tank to a pan filter (not shown), deliquored and fed by a feeder 1 into a stack 2, the alumina then being pneumatically conveyed through the stack 2 by outgoing hot gas from a duct 3. The alumina is dried by the outgoing hot gases and is then separated from those gases in a cyclone 10, while fluoride gas present in the outgoing hot gas is absorbed onto the surface of the alumina in the stack 2 and is thus returned to the process.

Gases separated in the cyclone 10 are forced through a duct 4 where they are mixed with high surface area smelter grade alumina dust which acts as a further scavenging agent for fluoride gas. The mixture then enters a multi-cyclone 5 where heavier particles pass down through the multi-cyclone, the particles being captured by the multi-cyclone 5 being useable in the calcining zone or the cooling zone (described below) as desired. The separated gases then pass via a duct 6 to a precipitator 7 wherein they are scrubbed and released to the environment through a stack 8.

The alumina captured in the cyclone 10 underflows from the cyclone 10 into a flash drier 20 through a feed duct 21. The flash drier 20 contains alumina that is fluidized by hot gases delivered thereto by a hot gas stream (not shown). Hot gases from the flash drier 20 feed via a duct 24 into the cyclone 10 wherein they mix with outgoing gases from the cyclone 10. The stack 2, the cyclone 10 and the flash drier 20 define, in combination, the dehydroxylation zone, all of which will be operated at essentially the same temperature, being in the range of 140°C to 180°C, but preferably being about 150°C.

The alumina underflows from the flash drier 20 through a duct 23 and is then pneumatically transferred via a hydrate conveying line (not shown) to a cross-over duct 30. Energy is exchanged between the outgoing hot gases from

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the holding vessel 40 and the alumina in the cross over duct 30. This begins the calcination process of the alumina.

The hot gases in the cross-over duct 30 carry the alumina into the cyclone 50 where the gases and the alumina are separated. The separated outgoing hot gases are forced from the cyclone 50 through the duct 3 where they recombine with fresh alumina entering through the feeder 1, while separated alumina underflows from the cyclone into a furnace 60.

In the furnace 60, fuel is burned in a series of burners directly into the calcining zone 61 which contains a suspension of alumina. The residence time of the alumina in the furnace 60 is short, and is preferably in the order of 10 to 100 seconds. The furnace 60 is preferably heated by combustion of gas where the furnace temperature is about 1150-1250°C. The furnace may however be heated by other fuels which would be known to a person skilled in the art, for example by combustion of fossil fuels or by electrical heating means.

In the present invention aluminium fluoride (preferably in an amount of about 5 kg/tonne of product) and excess steam are injected via a duct 62 into another duct 63 where they combine with outgoing hot gases from a pre-heat burner 70. The temperature in the duct 63 is about 1050°C. At this temperature the aluminium fluoride in the presence of excess steam undergoes rapid pyrohydrolysis, and the resultant excess hydrogen fluoride gas then enters the furnace 60 through its base to act as a mineralizing or catalytic agent to assist the transformation of alumina in the furnace to alpha alumina at a lower temperature than would otherwise be possible. The addition of aluminium fluoride immediately prior to the furnace 60 together with the excess steam maximises the yield of hydrogen fluoride and produces the greatest concentration of hydrogen fluoride in the area where the conversion to alpha alumina takes place.

The alumina gas suspension then passes from the furnace 60 into the cyclone portion (not shown) of the holding vessel 40, where solid alumina particles

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separated in the cyclone portion drop downwards into a fluidized bed within the holding vessel 40. Separated outgoing hot gases rich in hydrogen fluoride are then forced to the cross-over duct 30 where they meet with alumina from the hydrate conveying line.

- 5 The holding vessel 40 is maintained at a temperature of about 1250 to 1260°C. In this respect, alpha aluminas, and particularly A-12 type aluminas, are generally difficult to fluidize and this may be overcome by using tuyere-type distributor which provides a plurality of high velocity jets of air or steam through a distributor plate (not shown) in the base of the holding vessel 40 to fluidize the  
10 alumina.

The alumina is held in the fluidized bed in the holding vessel 40 for a period of approximately 1 to 50 minutes, and preferably 40 minutes, during which time it gradually works its way towards a transfer duct 41. It will be understood that the Residence time within the holding vessel will depend upon the temperature and  
15 the desired alpha alumina content of the product.

The fluidizing gas used in the holding vessel 40 is preferably steam as this promotes crystal growth and enhances conversion to alpha alumina. Usage of steam also allows operation of the holding vessel at elevated temperatures for long periods of time without collapse of the fluidized bed as a result of alumina  
20 particle stickiness at such temperatures. The steam may be superheated if desired.

Upon entering the transfer duct 41, the alpha alumina passes to a pneumatic conveyor line 80 where it meets cold air introduced through a duct 81. An energy exchange takes place resulting in the cooling of the alumina, following  
25 which the alumina travels along the conveyor line 80 until it reaches a further duct 82. Upon entering the duct 82 the alumina is driven into a cyclone 83 by outgoing gases from a cyclone 84.

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Solid alpha alumina particles pass down through the cyclone 83 while outgoing gases pass along a duct 85 into the pre-heat burner 70, and separated alpha alumina then passes via a transfer duct 86 to enter a duct 87 where it meets outgoing gases from the flash cooler 90. The alumina then enters cyclone 84  
5 whereafter separated alpha alumina passes down through the cyclone 84 while the separated outgoing gases are cyclonically driven along the duct 82 to collect alumina entering the duct via the conveyor line 80.

Separated alumina then passes via transfer duct 88 to a pneumatic conveyor line 91 wherein it combines with water and steam introduced through a duct 92.  
10 The alumina is then pneumatically conveyed to cyclone 93 such that separated alumina passes through the cyclone into the flash cooler 90, while outgoing gases pass through a duct 94 and meet up with separated alumina entering the duct 87.

Separated alumina entering the flash cooler is then cooled by heat exchange  
15 with incoming fluidizing air and water in heat exchange bundles prior to discharge onto a conveyer belt (not shown). The fluid bed cooler reduces the temperature of the product to less than 130°C prior to discharge.

The pneumatic conveyor line 80, the cyclone 83, the cyclone 84, the pneumatic conveyor line 91, the cyclone 93 and the flash cooler 90 define, in combination,  
20 the cooling zone. The alumina product on entering this zone is extremely friable and may be excessively broken down by high velocities in the transfer ducts and cyclones. To minimize the gas volumes and temperatures in the flash cooling section (and hence minimize velocities) water can be sprayed into the ducts 82 and the conveyor line 91 and steam can be introduced into conveyor line 91.  
25 The proportions of steam and water introduced into the duct and line are preferably ratioed to balance the thermal mass required to dry the product in the stack 2 in the dehydroxylation zone. This balance may be formulated by using a computer and mass balance model which would be known to a person skilled in the art. The use of water and steam removes the requirement to use excess  
30 air in the flash cooler simply for cooling the outgoing product.

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It will be appreciated that there may be further variations and modifications which will be apparent to those skilled in the art from the teaching of the above, and such variations and modifications are also deemed to be within the scope of the invention as herein disclosed.



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The Claims defining the invention are as follows:

1. A process for the production of alpha alumina, the process comprising the steps of:
    - 5 (a) drying and dehydrating alumina in a dehydroxylation zone to remove residual water;
    - (b) heating the dehydrated alumina in a calcining zone in the presence of hydrogen fluoride gas to a temperature above that in the dehydroxylation zone to commence transformation of the alumina to alpha alumina; and
    - 10 (c) holding the heated alumina in a holding zone for sufficient time and at a sufficient temperature to complete the transformation of the alumina to alpha alumina;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone.

  - 15
2. A process according to claim 1 wherein steam is added to the aluminium fluoride at its point of addition to the calcining zone to ensure the immediate generation of excess hydrogen fluoride.
  3. A process according to claim 1 wherein the temperature in the dehydroxylation zone is in the range of 100°C to 800°C.
  - 20
  4. A process according to claim 3 wherein the temperature in the dehydroxylation zone is about 150°C.
  5. A process according to claim 1 wherein the heating of the dehydrated alumina in step (b) is conducted in a furnace.

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- 6 . A process according to claim 5 wherein the aluminium fluoride is injected at or near the base of the furnace at a region where the temperature is above 1000°C.
- 7 . A process according to claim 1 wherein the alumina is heated in the  
5 calcining zone at a temperature in the range of 1120°C to 1280°C.
- 8 . A process according to claim 7 wherein the alumina is heated in the calcining zone at a temperature of about 1200°C.
- 9 . A process according to claim 7 wherein the residence time is in the range of 10 to 100 seconds.
- 10 10 . A process according to claim 1 wherein the holding zone of step (c) includes a fluidised bed.
- 11 . A process according to claim 10 wherein the heated alumina is maintained in the fluidised bed for 30 to 45 minutes.
- 12 . A process according to claim 10 wherein the heated alumina is  
15 maintained in the fluidised bed at a temperature in the range of 1200°C to 1300°C.
- 13 . A process according to claim 1 wherein the dehydrating step of step (a) produces dehydrated alumina and waste gases, the waste gases being mixed with a stream of smelter grade alumina dust.
- 20 14 . A process according to claim 13 wherein the heating of the dehydrated alumina in step (b) produces outgoing hot gases used in the dehydroxylation zone of step (a).
- 15 . A process according to claim 1 including the step of cooling the alpha alumina produced in step (c) in a cooling zone such that the temperature of the  
25 alpha alumina is reduced to less than 200°C prior to discharge.

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16. A process according to claim 1 substantially as herein described in relation to the accompanying drawing.
17. An alpha alumina produced by a process in accordance with any one of claims 1 to 16.
- 5 18. A process for the production of alpha alumina, the process comprising the steps of:
- (a) drying and dehydrating alumina in a dehydroxylation zone at a temperature in the range of 100°C to 800°C to remove substantially all residual water ;
  - 10 (b) heating the dehydrated alumina in a calcining zone within a furnace, in the presence of hydrogen fluoride gas, to a temperature in the range of 1120°C to 1280°C to commence transformation of the alumina to alpha alumina;
  - 15 (c) holding the heated alumina in a holding zone within a fluidised bed for 30 to 45 minutes at a temperature in the range of 1200°C to 1300°C to complete the transformation of the alumina to alpha alumina; and
  - 20 (d) cooling the alpha alumina in a cooling zone where the temperature of the alpha alumina is reduced to less than 200°C prior to discharge ;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone, steam being added to the aluminium fluoride at that point to ensure the immediate generation of excess hydrogen fluoride.

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19. A process according to claim 18 substantially as herein described in relation to the accompanying drawing.

20. An alpha alumina produced in accordance with the process of either of claims 18 or 19.

5 21. A process for the production of alpha alumina, the process comprising the steps of:

- 10 (a) drying and dehydrating alumina in a dehydroxylyzation zone at a temperature in the range of 100°C to 800°C to remove substantially all residual water, thereby producing dehydrated alumina and waste gases, said waste gases being mixed with a stream of smelter grade alumina dust to remove fluoride introduced by the outgoing hot gases from step (b);
- 15 (b) heating the dehydrated alumina in a calcining zone within a furnace, in the presence of hydrogen fluoride gas, to a temperature in the range of 1120°C to 1280°C to commence transformation of the alumina to alpha alumina, and thereby generating outgoing hot gases for use in the dehydroxylyzation zone of step (a);
- 20 (c) holding the heated alumina in a holding zone within a fluidised bed for 30 to 45 minutes at a temperature in the range of 1200°C to 1300°C to complete the transformation of the alumina to alpha alumina;
- 25 (d) cooling the alpha alumina in a cooling zone where the temperature of the alpha alumina is reduced to less than 200°C prior to discharge, thereby producing a cooled alpha alumina product and outgoing gases ; and

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- (e) heating the cooling zone outgoing gases for return to the calcining zone of step (b) ;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone, steam being added  
5 to the aluminium fluoride at that point to ensure the immediate generation of excess hydrogen fluoride.

22 . A process according to claim 21 substantially as herein described in relation to the accompanying drawing.

23 . An alpha alumina produced in accordance with the process of either of  
10 claims 21 or 22.

24 . Apparatus for the production of alpha alumina, the apparatus comprising :

- (a) means for drying and dehydrating alumina in a dehydroxylation zone to remove residual water;
- 15 (b) means for heating the dehydrated alumina in a calcining zone in the presence of hydrogen fluoride gas to a temperature above that in the dehydroxylation zone to commence transformation of the alumina to alpha alumina; and
- 20 (c) means for holding the heated alumina in a holding zone for sufficient time and at a sufficient temperature to complete the transformation of the alumina to alpha alumina;

wherein the hydrogen fluoride gas in step (b) is generated by pyrohydrolysis of aluminium fluoride at or near the entry to the calcining zone.

25 . Apparatus according to claim 24 wherein the means for holding the heated alumina includes a fluidised bed within which the alumina may be held  
25 for the sufficient time and temperature.

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26 . Apparatus according to claim 25 wherein the fluidised bed includes a tuyere-type distributor plate at the bottom thereof to assist in fluidising the alpha alumina.

27 . Apparatus according to claim 24 substantially as herein described in  
5 relation to the accompanying drawing.

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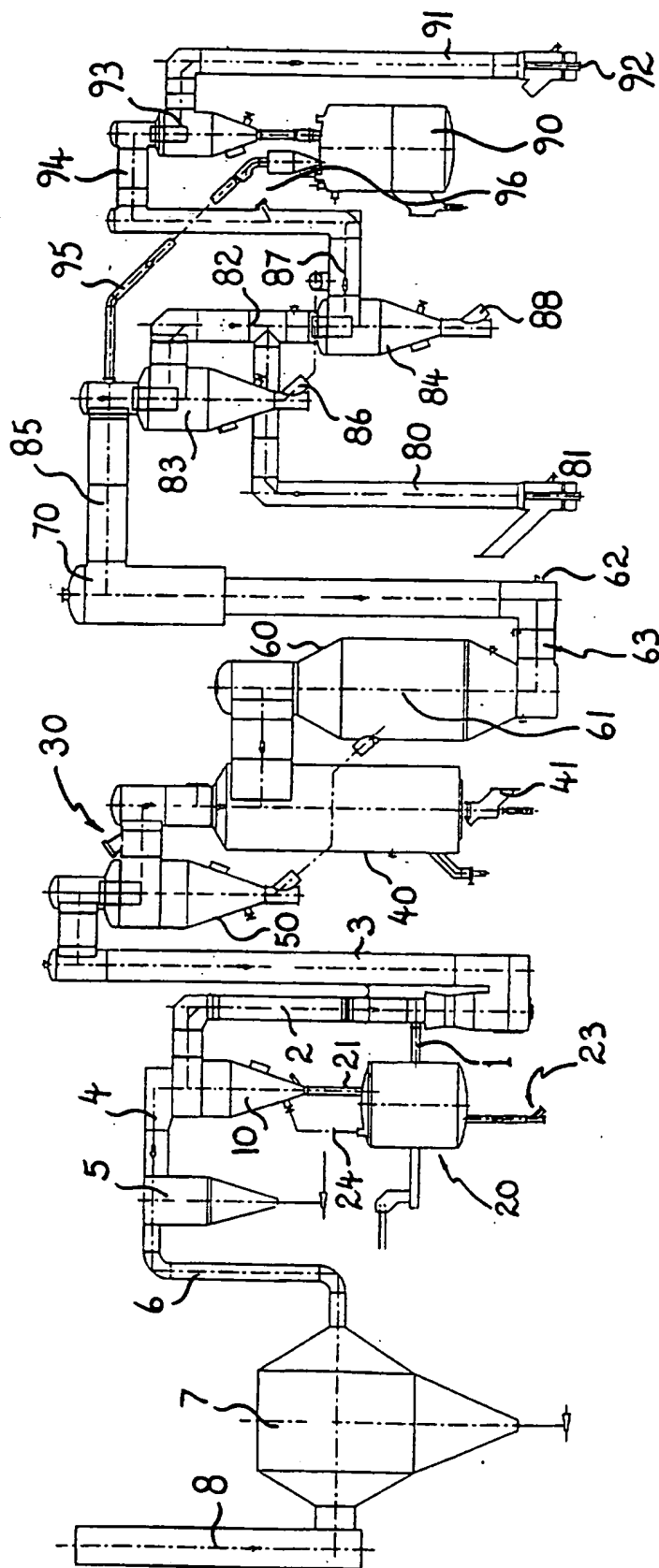


Fig. 1

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/AU 96/00011

## A. CLASSIFICATION OF SUBJECT MATTER

Int Cl<sup>6</sup>: C01F 7/22

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC : C01F 7/22

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
AU : IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
DERWENT

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 620188 <u>A1</u> (SUMITOMO CHEM CO LTD) <u>19 October 1994</u>	
A	Derwent Abstract Accession No. 93-197667/25, Class E33, CN, A, 1065255 (LIB) 24 October 1992	
A	AU 585568 <u>A1</u> (MONTEDISON S.P.A.) <u>24 July 1986</u>	



Further documents are listed in the continuation of Box C



See patent family annex

### \* Special categories of cited documents:


- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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- "&" document member of the same patent family

Date of the actual completion of the international search  
20- April 1996

Date of mailing of the international search report  
24-04-96

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# INTERNATIONAL SEARCH REPORT

## Information on patent family members

International Application No.  
PCT/AU 96/00011

**This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.**

Patent Document Cited in Search Report				Patent Family Member			
EP	620188	AU	59410/94	CA	2121198	JP	7206430
AU	5242686	CA	1288575	EP	188388	US	4818515

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